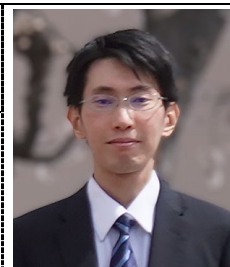


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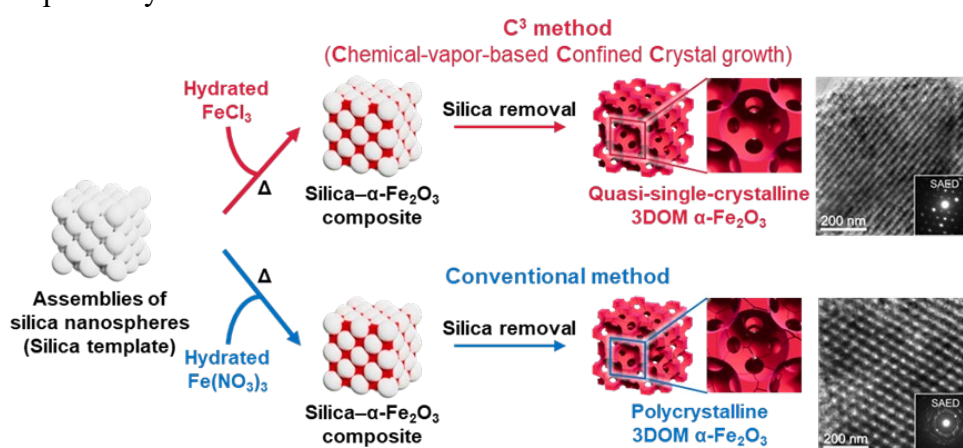
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Synthesis of Quasi-Single Crystalline Nanoporous Iron Oxides using Metal Chloride as a Precursor

Ordered nanoporous metal oxides have attracted attention for their broad applications across fields, including catalysts, energy materials, and biomaterials. In addition to the compositions and porous structures, controlling the crystallite size of the pore walls is crucial for improving performance.¹ However, precise control of the nanostructures from the atomic to the nano-scale range is still challenging, because of the limitation of the conventional synthesis method.

In this study, we report the preparation of a quasi-single-crystalline nanoporous metal oxide via oxidation of a metal chloride precursor within the nanopores of silica templates (Scheme 1).² We focused on iron oxide, which is used in various applications such as catalysts and electrodes, and investigated its properties in an inverse opal (3DOM) structure with a high specific surface area. Iron chloride inside the template is transported in the vapor phase upon heating, enabling the nucleation and crystal growth of α -Fe₂O₃. As a result, nanoporous α -Fe₂O₃ with a larger and more uniform crystallite size than that prepared using the conventional Fe(NO₃)₃ hydrate precursor was achieved. The quasi-single-crystalline nanoporous α -Fe₂O₃ exhibited higher catalytic activity and thermal stability than the conventional nanoporous α -Fe₂O₃ composed of nanocrystals, as confirmed by methylene blue degradation and heating tests, respectively.



Scheme 1. Synthesis of 3DOM α -Fe₂O₃ with different crystallite sizes.

References: (1) W. Li and K. Xie, *Acc. Chem. Res.*, **56**, 374–384 (2023). (2) D. Oka, K. Takaoka, A. Shimojima, and T. Matsuno, *Chem. Mater.*, 10.1021/acs.chemmater.5c00155 (2025).